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### A METALLOGRAPHIC STUDY OF WHITE LAYERS IN GUN STEEL

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August 1978



# US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND LARGE CALIBER WEAPON SYSTEMS LABORATORY BENÉT WEAPONS LABORATORY WATERVLIET, N. Y. 12189

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A metallographic investigatio	n has been made	of the white layers formed on	
the bore surface of a fired Army a	nd Navy cannon a	nd those produced in gun steel	
specimens in laboratory where firing conditions were simulated. White layers			
are produced in laboratory specime	ns in reducing e	nvironments (e.g. methane gas)	
but not in argon or nitrogen and a	ppear similar to	those produced in the fired	

cannons. These are formed at the melting as well as lower temperatures. The

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effects of increase in the pressure of the environment appears first to aid the formation of white layers and furthermore to increase their thickness. These results and the earlier studies of the characterization of white layers from fired cannons suggests that carbon from the gaseous environment of the propellant combustion products and the pressures of gases have significant effects on the formation and growth of white layers.

The white layers produced in the Navy cannon where NACO, low flame temperature propellant was used are compared with those produced in the Army cannons where high flame temperature propellant was used. These observations are also discussed.

#### **ACKNOWLEDGEMENTS**

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#### TABLE OF CONTENTS

		Page
ACKI	NOWLEDGEMENTS	i
INT	RODUCTION	1
PRO	CEDURE	2
A	. Material	2
В	. Sample Preparation and Examination	3
RES	ULTS	3
Α	. The Five-Inch Navy Cannon	3
В	. The 105mm and 155mm Army Cannons	4
С	. The Laboratory Tested Samples	5
DIS	CUSSION	5
REF	ERENCES	10
	ILLUSTRATIONS	
1.	Cracking and "white layer" observed along bore of a rapid-fired Navy gun.	11
2.	Formation of "white layer" at the tip of cracks running through the chrome plating. Note absence of "white layer" where chrome is intact.	11
3.	Microhardness of the four individual layers which comprise the "white layer" along the bore of a rapid-fired Navy gun.	12
4.	Typical tempered martensite structure in steel.	12
5.	"White layer" observed along bore of 105mm Army cannon.	13
6.	Average of several microhardness readings taken in the individual layers comprising the "white layer" along the bore of 105mm cannon.	13

		Page
7.	Laboratory produced "white layer". Specimen was tested in methane gas at 1000 psi. Note the similarity in microstructure and microhardness of the various layers to those of the fired cannons.	14
8.	(a) Specimen tested at low pressures (500 Torr) in methane gas. The resulting thin white layer is typical of low pressure tests. However, occasional relatively larger localized white layer areas are produced and one is shown, along with microhardness indentations, in Figure 8(b).	15
9.	Shows various white layers, their microhardness and the region where melting occurred in laboratory specimen tested in methane gas at 1000 psi pressure.	16
10.	Typical white layer formed in high pressure methane gas when a specimen which was taken considerable distance away from the melted region and also when a specimen was heated below the melting temperature of steel and in which no melting had occurred.	16
11.	A specimen such as the one in Figure 10 shows various white layers formed and their microhardnesses.	17

#### INTRODUCTION

During firing, the bore surface of the cannon is subjected to severe conditions of a short, high temperature, high pressure pulse in an environment of the propellant gases, consisting primarily of carbon monoxide, carbon dioxide, methane, nitrogen, water vapor, sulfur dioxide, ammonia and others. These conditions are most severe in the origin of rifling region and appear responsible for the formation of the so-called white surface layers found almost exclusively on the bore surface of this region. Elsewhere, heat affected zones are produced whose microstructures suggest those of tempered martensite. An investigation of the processes that lead to the formation of white layers and the chemical and microstructural characteristics of these layers may aid in an improved understanding of the wear and erosion phenomena in cannons.

The purpose of this investigation is to characterize and compare, using metallographic techniques, the white layers formed on cannons with those produced on laboratory samples tested under conditions simulating firing and in selected gaseous atmospheres.

<sup>&</sup>lt;sup>1</sup>"Gun Tube Erosion and Corrosion," Proc. Interservice Tech. Meeting, Editors, I. Ahmad and J. P. Picard, February 1970.

<sup>&</sup>lt;sup>2</sup>"Metallurgical Examination of Bore Surface Damage in a 5 Inch Gun," R. B. Griffin, C. Morris and J. Pepe, Watervliet Arsenal Technical Report WVT-TR-74028, July 1974.

#### PROCEDURE

#### A. Material

Specimens for metallographic study were taken from three sources.

- (1) Specimens were cut from the origin of rifling of a five inch Navy cannon which was rapidly test fired a total of 587 rounds in 82 minutes, using a NACO propellant having a flame temperature of 2200°K.
- (2) Specimens were taken from the origin of rifling of a 105mm and a 155mm Army cannon. Each was fired a total of 1510 rounds during their service life using a M30-Al propellant having a flame temperature of 3040°K.
- (3) Specimens of material taken from cannon barrels were tested in the laboratory under conditions simulating firing. A pulse heating apparatus available at the Benet Laboratory<sup>3</sup> was modified and used in both low and high pressure tests and in various gaseous atmospheres, such as carbon monoxide, methane, carbon dioxide, and different ratios of carbon monoxide and carbon dioxide. Some specimens were heated in various environments until localized incipient melting occurred and they broke into two pieces. From the broken pieces, specimens were taken (a) where melting occurred and (b) away from the melted region. In other studies, test specimens were heated below the melting temperature

<sup>&</sup>lt;sup>3</sup>"The Pulsar - An Ultra High Speed Heating and Quenching System," Watervliet Arsenal Technical Report WVT-7250, September 1972.

of steel and a section was cut from these unbroken specimens. These specimens were used for metallographic examinations.

#### B. Sample Preparation and Examination

Small specimens were cut from the areas of interest and mounted at a slight taper (approximately 15°) to spread out the thin surface layers. The specimens were mounted in a polyester embedding material mixed with an aluminum oxide hardness equalizer to prevent rounding of the edges of the specimens during polishing. Initially, the specimens were rough polished using standard metallographic procedures. They were subsequently polished using paper embedded with diamond paste which provided better edge retention for surface studies than could be obtained using the standard polishing cloth. Finally, the specimens were polished in a slurry of distilled water and 0.3 micron alumina using a "Syntron" vibratory polisher.

The specimens were etched with either picral plus hydrochloric acid or superpicral etchants to reveal the microstructure of the white layers and the matrix material. The specimens were examined metallographically and, using a 25 gram load, Knoop microhardness indentations were made in the surface layers.

#### RESULTS

#### A. The Five-Inch Navy Cannon

Specimens taken from the rapid-fired Navy gun show large amounts of various white layers and much cracking. A typical example is shown in Figure 1. It should be noted that the heat affected zones do form under the chrome plating however, white layers did not form

unless the chrome plating was cracked or missing. Figure 2 shows white layers beginning to form at the tip of a crack running through the chrome plating and the absence of the layer where the chrome is intact. The four layers which comprise the so-called "white layer" are observed on the samples and their hardness characteristics are shown in Figure 3. The outer layer was fairly hard (KHN 400-500) and appears to have some substructure. The second layer was somewhat harder (KHN 550-650) while the third, or usually featureless, nonetching layer was quite soft (KHN 300-400). The heat affected zone which appears to be tempered martensite structure had a Knoop hardness of about 700 where as the martensite microstructure of the typical gun steel shown in Figure 4 had a microhardness of ~ 400 (KHN).

#### B. The 105mm and 155mm Army Cannons

The various white layers found on the specimens taken from fired guns are shown in Figure 5. The presence of all the layers at a specific area in the specimens occurred but infrequently compared to those observed in the rapid-fired Navy cannons. Also, the layers were a great deal thinner; compare Figures 3 and 5. The more predominant nonetching white layers formed on these cannons were soft, Figures 5 and 6. Both the microhardness and the microstructure of the white layers on the Army cannon specimens are similar to those on the Navy cannon specimens; compare Figures 3 and 5. The formation of the various white layers and their increased frequency of occurrence may be more related to the very rapid rate of firing than to the type of propellant used.

#### C. The Laboratory Tested Samples

The specimens tested in methane gas at 1000 psi produced white layers which are shown in Figure 7. The microhardness and microstructures of these layers are similar to those of corresponding layers of the Army and Navy cannons previously discussed; for comparison, see Figures 3, 5, 7, 8, 9, 10, and 11.

It was noted that the white layers formed in high pressure gases were thicker and can be reproduced in just about every test whereas these were thinner and formed infrequently when tested at low pressure 500 Torr (~ 8 Psi) in methane gas and in methane and carbon dioxide mixtures. The relative thickness of these layers can be seen readily by comparing Figures 7 and 8. Apparently, pressure has some effect in the stabilization and growth of white layers. In addition, it should be noted that white layers do form at and below the melting temperature of steel, Figures 8-10. Thus, both the pressure and the temperature have significant effects in the formation of white layers.

In other atmospheres, such as nitrogen or argon, the specimens tested at 1000 psi or 500 Torr (~ 8 psi) did not form white layers. However, heat affected zones were observed.

#### DISCUSSION

The microstructure and microhardness of the white layers formed on the specimens taken from fired cannons and laboratory test specimens are quite similar; see Figures 3, 5, 7, 8 - 11. Furthermore, the white layers in the laboratory test specimens form only in reducing carbonaceous atmospheres such as methane and methane-CO<sub>2</sub>

gas mixtures and not in nitrogen, argon, or CO2. Earlier investigations to characterize the white layers formed in a cannon using Auger, ESCA, x-ray diffraction, etc., indicate that the white layers are high carbon austenite (soft white layer) and ε-carbide (hard white layer). 4 This suggests that carbon from reducing atmospheres is responsible for the formation of these layers and provides support for the idea that the white layers formed in the laboratory are the same or similar to those formed on the bore surface of fired cannons. It is significant to note that white layers do form at and below the melting temperature of steel and their characteristic features and microhardnesses are similar to the white layers formed in the fired cannon, Figure 8 - 10. These observations suggest that interaction of reducing atmospheres with heated steel produces carbon which is incorporated in steel via solid state diffusion. The high heating and cooling rates then lead to the formation of white layers which are believed to be non-equilibrium phases.

Of specific interest are the effects of pressure of the environment on the formation and growth of white layers produced in laboratory specimens 'pulse' heated under identical conditions. An increase in pressure of methane gas from 8 psi (500 Torr) to 1000 psi - two orders of magnitude increase - virtually assures the formation of soft white layers and significantly increases the thickness of the layers

<sup>&</sup>lt;sup>4</sup>"Characterization of Bore Surfaces in Large Gun Barrels," J. Venables MML TR-76-55C, July 1976, report submitted to M. H. Kamdar, Watervliet Arsenal, Watervliet, NY.

formed from those produced at low pressures (8 psi), compare Figures 7 and 8. These effects may be explained as follows. At higher pressures, the increased possibility of interaction may increase the amounts of carbon produced and its diffusion in the specimen surface at elevated temperature, increasing thereby the carbon content of the surface layers. Increased carbon content and high temperatures promote growth of austenite while fast cooling rate promote its retention as a non-equilibrium phase at low temperatures. This appears reasonable, since the soft white layers formed in the bore surface of the fired cannon are in fact shown to be high carbon austenite.

The above discussion suggests the following conditions for the formation and the growth of white layers:

- 1. A specific carbon containing reducing environment is required.
- 2. The growth kinetics are controlled by the temperature and pressure which produce carbon and via diffusion increase the carbon content of the surface layers of the specimen.
- 3. Fast cooling rates are required to retain high carbon high temperature phases, i.e., white layers at lower temperatures.

  This suggests that carbon producing reducing gases in the combustion products of the propellant and the temperature and the pressure of these gases may have significant effects on the formation and growth of white layers in the fired cannons.

<sup>&</sup>lt;sup>4</sup>"Characterization of Bore Surfaces in Large Gun Barrels," J. Venables MML TR-76-55C, July 1976, report submitted to M. H. Kamdar, Watervliet Arsenal, Watervliet, NY.

The white layers are always produced in both the Navy and the Army cannons which experience about the same pressures (40 to 50 Ksi) upon firing but use propellants with a low (2200°K) and a high (3040°K) flame temperature for the Navy and the Army cannons respectively. In Navy cannons, the first white layer formed is hard and the second white layer formed is soft whereas in the Army cannon the first and the most predominantly observed white layer is the soft white layer, Figures 1, 5 and 6. It appears that the extremely rapid test firing rate experienced by the Navy cannon compared to the intermittent conventional firing of Army cannon in service over a prolonged period of use may be the reason for the observed behavior. At very rapid firing rates, the bore surface of the cannon will remain at an elevated temperature for a longer time period thereby increasing the amounts of carbon produced via the interaction of gaseous species with steel. Carbon will diffuse in the bore surface of the cannon producing a concentration gradient with increased carbon content in the immediate surface layers and then decreasing carbon content with depth from the surface. High carbon content in the immediate bore surface layers may lead to the formation of hard high carbon (~ 10% carbon) containing carbides phase or needles and then the soft low carbon containing (~ 1% carbon) austenite phase 4 in the subsequent layer as seen in Figure 1. On the other hand, based on the same reasoning as above, intermittent firing

<sup>&</sup>lt;sup>4</sup>"Characterization of Bore Surfaces in Large Gun Barrels," J. Venables MML TR-76-55C, July 1976, report submitted to M. H. Kamdar, Watervliet Arsenal, Watervliet, NY.

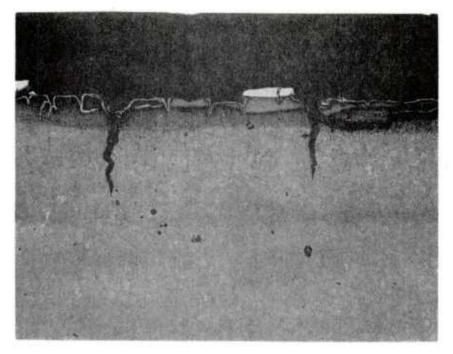
of Army cannon may result in lower carbon content in the surface layers promoting the formation of soft white layers. Thus, the variation in the observed behavior in the Navy and Army cannons is most likely caused by the firing rate rather than the flame temperature of the propellant used, i.e., by the time at temperature that the environment interacts with the gun steel. Possibility exists though that besides rapid firing, the presence of chrome plating affecting the heating and cooling rates, etc., and the absence of chrome plating on the Army cannon studied may also be important in the order in which these layers form. It is apparent from this investigation however, that a specific environment and its pressure and temperature have significant effects on the formation and the growth of various white layers in gun steel.

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- "Gun Tube Erosion and Corrosion," Proc. Interservice Tech Meeting,
   Editors, I. Ahmad and J. P. Picard, February 1970.
- "Metallurgical Examination of Bore Surface Damage in a 5 Inch Gun,"
   R. B. Griffin, C. Morris, and J. Pepe, Watervliet Arsenal
   Technical Report WVT-TR-74028, July 1974.
- 3. "The Pulsar An Ultra High Speed Heating and Quenching System," Watervliet Arsenal Technical Report WVT-7250, September 1972.
- 4. "Characterization of Bore Surfaces in Large Gun Barrels," J.

  Venables MML TR-76-55C, July 1976, report submitted to M. H. Kamdar,

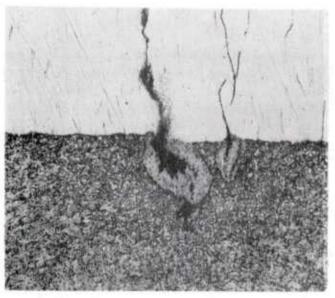
  Watervliet Arsenal, NY.



Mag. 50x

Etchant: 2% Picral + HC1

Figure 1. Cracking and "white layer" observed along bore of a rapid-fired Navy gun.

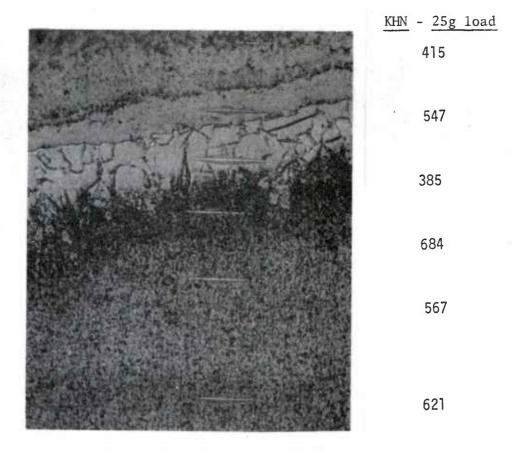


Chrome Plate

Altered Structure

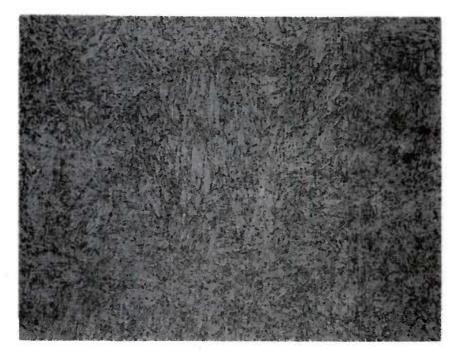
Mag. 750x Etchant: 2% Picral + HC1

Figure 2. Formation of "white layer" at the tip of cracks running through the chrome plating. Note absence of "white layer" where chrome is intact.



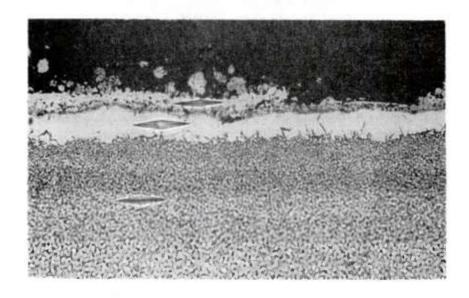
Mag. 1500X Etchant: Super Picral

Figure 3. Microhardness of the four individual layers which comprise the "white layer" along the bore of a rapid-fired Navy gum.



Mag. 1000X Etchant: 2% Picral + HC1

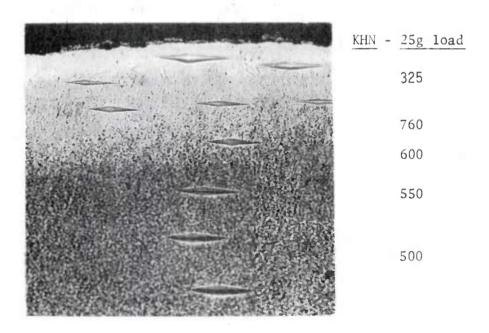
Figure 4. Typical tempered martensite structure in steel.



Mag. 500X

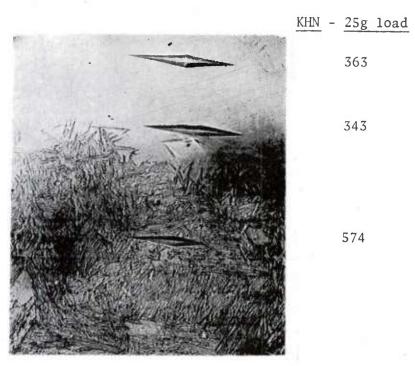
Etchant: 2% Picral + HC1

Figure 5. "White layer" observed along bore of 105mm Army cannon.



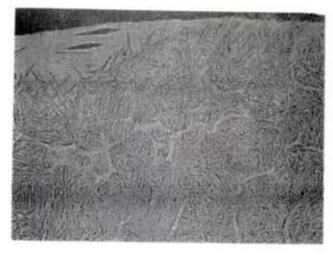
Mag. 500X Etchant: 2% Picral + HC1

Figure 6. Average of several microhardness readings taken in the individual layers comprising the "white layer" along the bore of a 105mm cannon.

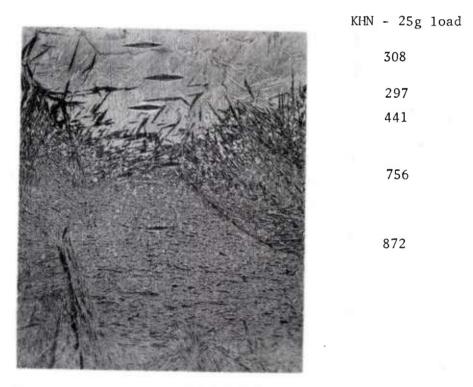


Mag. 750X Etchant: 2% Picral + HCl

Figure 7. Laboratory produced "white layer". Specimen was tested in methane gas at 1000 psi. Note the similarity in microstructure and microhardness of the various layers to those of the fired cannons.

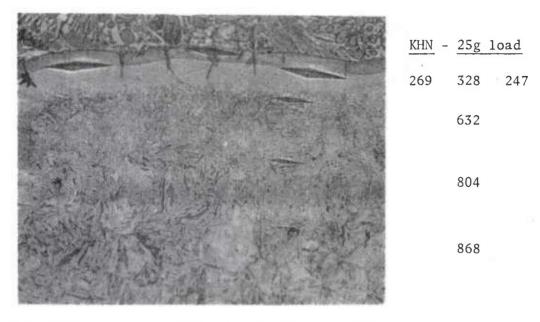


a) Mag. 500X Etchant: 2% Picral + HC1



b) Mag. 300X Etchant: 2% Picral + HCl

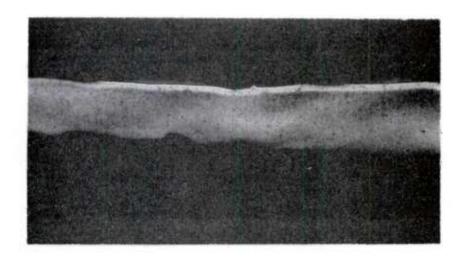
Figure 8: (a) Specimen tested at low pressures (500 torr) in methane gas. The resulting thin white layer is typical of low pressure tests. However, occasional relatively larger localized white layer areas are produced and one is shown, along with microhardness indentations, in Figure 8b.



Mag. 500X

Etchant: 2% Picral + HCl

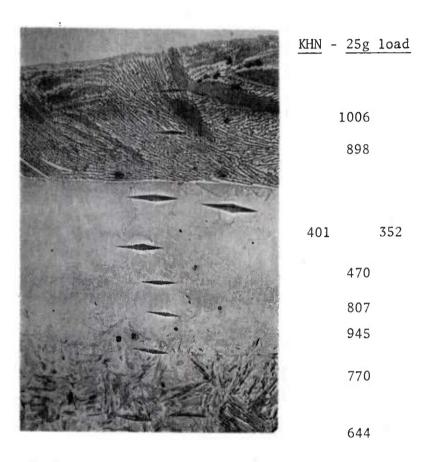
Figure 9. Shows various white layers, their microhardness and the region where melting occurred in laboratory specimen tested in methane gas at 1000 psi pressure.



Mag. 20X

Etchant: 2% Picral + HCl

Figure 10. Typical white layer formed in high pressure methane gas when a specimen which was taken considerable distance away from the melted region and also when a specimen was heated below the melting temperature of steel and in which no melting had occurred.



Mag. 500X Etchant: 2% Picral + HCl

Figure 11. A specimen such as the one in Figure 10 shows various white layers formed and their microhardnesses.

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